Fabrication and Operando Measurements of Optical and Spectro-electrochemical Cells for Battery Analysis

Jonathan Buntinx
Master of Energy Engineering Technology

Martijn Schepers
Master of Electromechanical Engineering Technology

Situation

This thesis is conducted within the research group imo-imomec, a collaboration between IMEC and Hasselt University. The Electrochemical Engineering group within imo-imomec specialises in electrochemical systems such as fuel cells and advanced batteries.
Lithium-based batteries, and more specifically lithium-ion batteries are dominant in the energy storage sector due to their energy density and cycle life, but they undergo internal changes during operation. Understanding these processes benefits from operando techniques. Optical operando analysis provides a way to observe changes without disassembling the cell and is particularly valuable because it captures behaviour under realistic working conditions. In this thesis, such a optical cell is designed and manufactured.

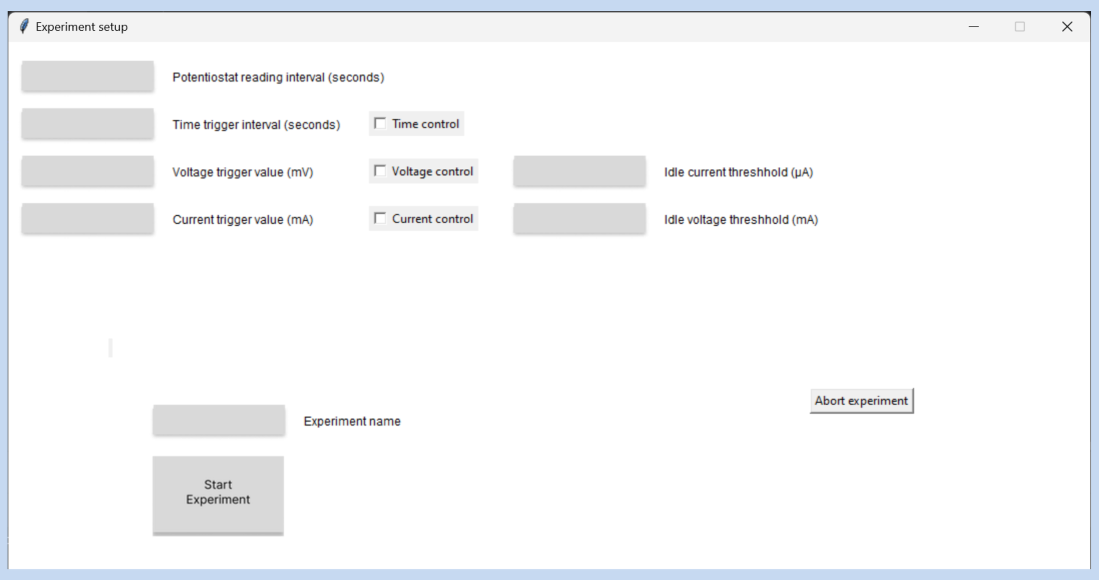

Figure 2: GUI of custom software that controls the digital microscope

Method

The project was carried out in four main phases:
Design: Several CAD iterations were made to integrate pressure control, electrical contacts, and a clear optical path. Constraints included working distance of objectives, sealing reliability, and straightforward operation in a glovebox.
Fabrication: Components were CNC-machined at the UHasselt workshop. Several design modifications were made during production.
Verification: Functional prototypes were assembled in a glovebox. Testing included checking the seal integrity, visibility, and coherence of the assembly.
Analysis Setup: Custom software was written to synchronise electrochemical data with optical image capture, menu is illustrated in figure 2.
Raman measurements and time-lapse microscopy were performed to observe phenomena for lithium/sulfur assemblies.

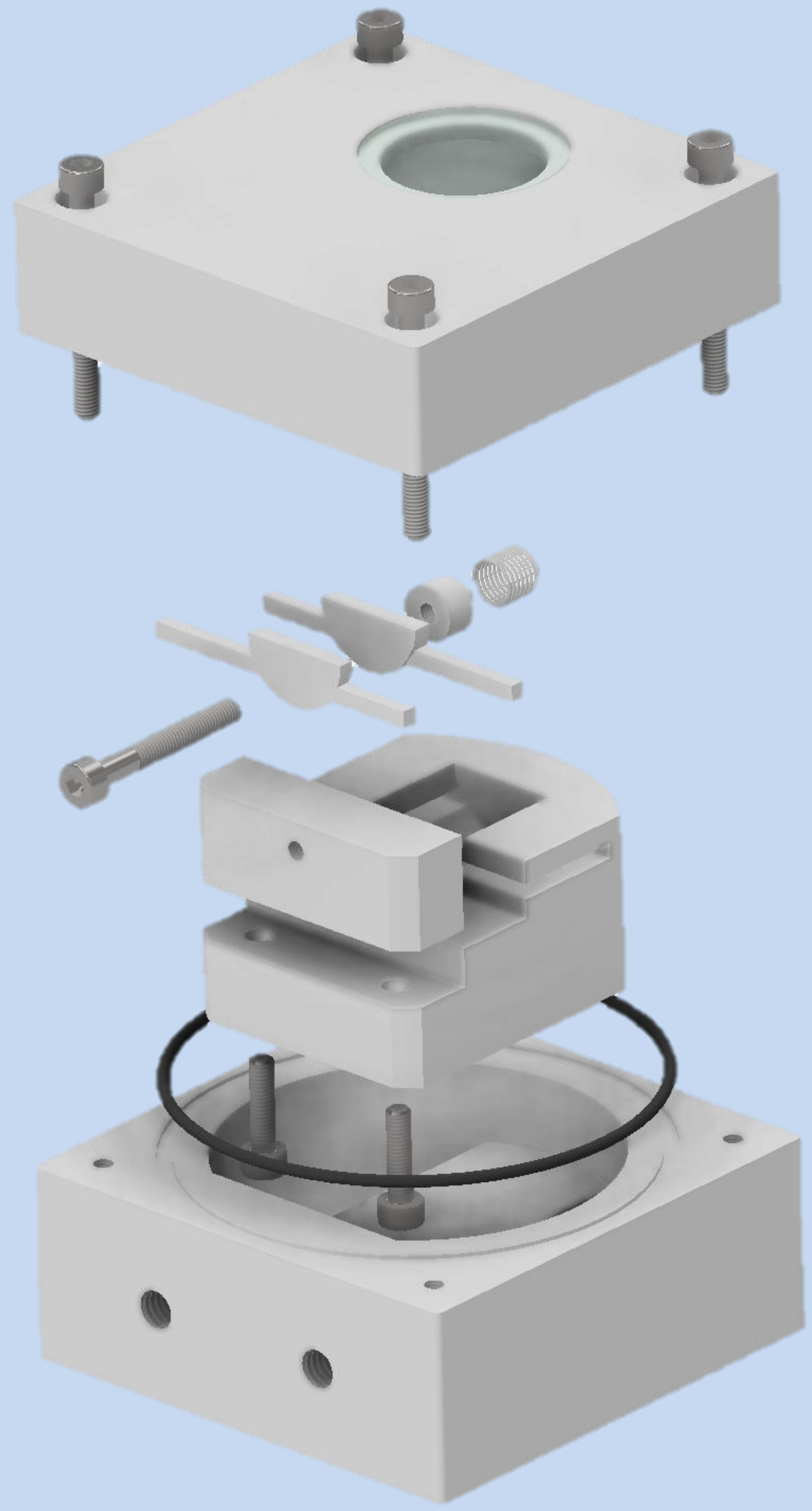

Figure 1: Opticall cell assembly

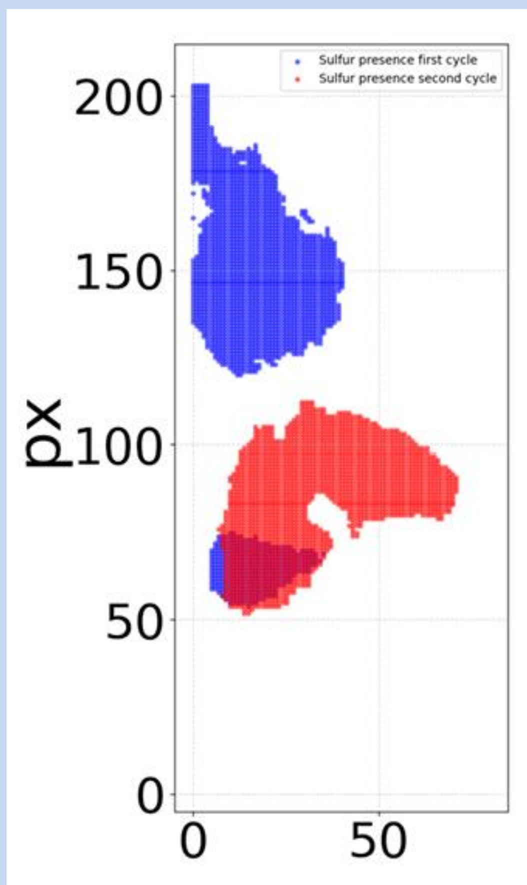

Figure 4: Sulfur presence at the electrode cross section surface: pristine state (blue), after one cycle (red)

Objectives

The goal is to design and fabricate a reusable and optically accessible battery cell that allows for observation of the cross-section of a halved coin cell battery stack
Requirements include:

- Chemically inert and pressure-stable enclosure.
- Compatible with both optical microscopy and Raman spectroscopy.
- Enable successful cycling without short circuits or leaks.
- Maintain good visibility of the electrode cross-section.
- Control of pressure on the battery stack

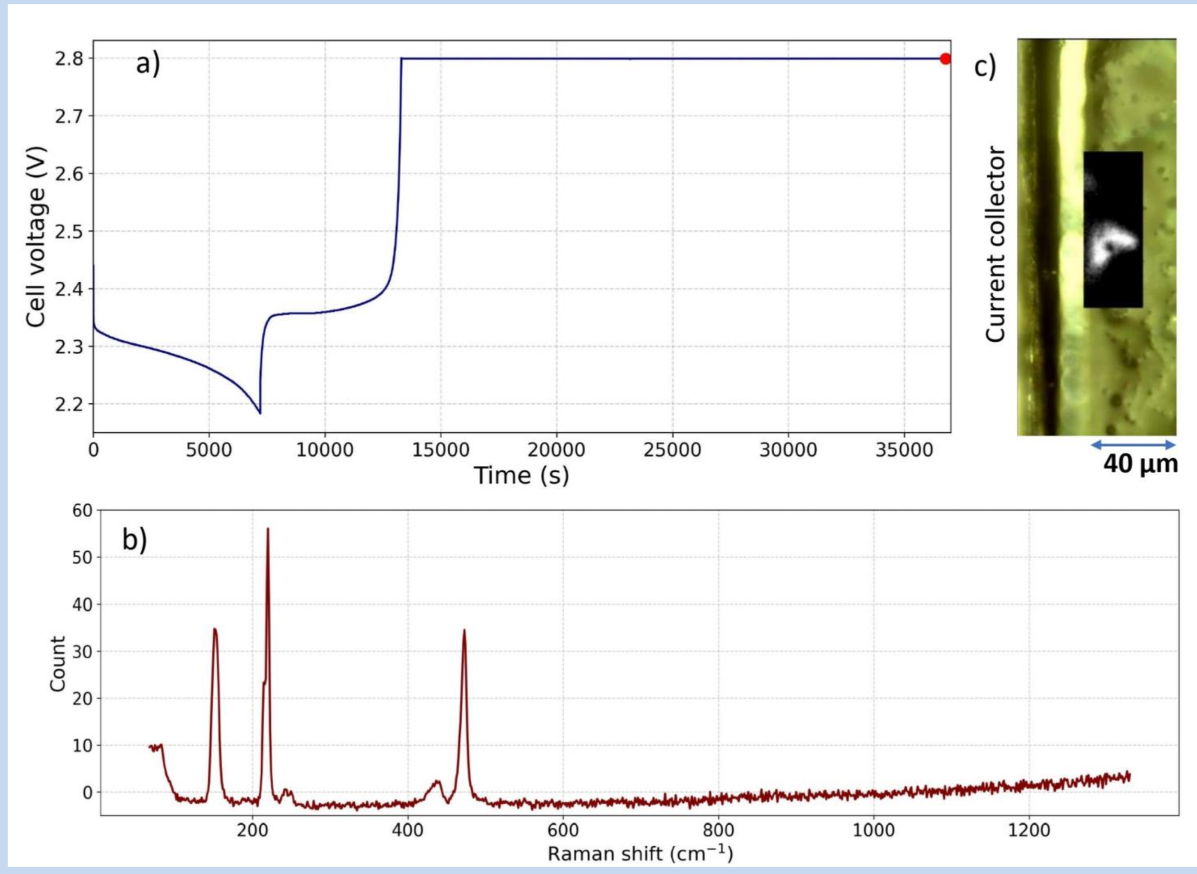

Figure 2: a) Raman shift, b) voltage curve and time curve, c) Raman mapping area

Results

The optical cell visible in figure 1, has these components from top to bottom beginning with a sapphire spectroscopic window mounted on the stainless steel top cover, followed by compression springs, nut caps, sliders for the working and lithium electrodes, a threaded bolt block for the compression bolt, a PEEK cell holder, a Viton O-ring for sealing, and the stainless steel bottom enclosure with electrical pass-throughs. The final prototype enabled optical and Raman measurements during battery cycling. Optical microscopy confirmed graphite expansion and sulfur dissolution. In Raman tests, sulfur species could be tracked spatially and temporally , revealing a dissolution gradient In figure 3, (a) the Raman shift indicates sulfur presence, (b) potential curve visualises the successful cycle. (c) Mapping of the sulfur electrode given after one cycle, ending up with the red particle visible in figure 3.

Conclusion

The optical cell works and is reusable with straightforward assembly. Sulfur transport was successfully observed during cycling. Some mechanical parts could be improved, but the design is a strong starting point for future studies for different cell chemistries.

Supervisors / Co-supervisors / Advisors: prof. dr. ir. Mohammadhosein Safari
dr. Saeed Yari
Albin Conde Reis